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Synthesis of Highly Porous Al₂O₃-YAG Composite Ceramics

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Abstract:

 Al_2O_3 -YAG composite was obtained by sintering of porous Al_2O_3 preforms infiltrated with water solution of aluminium nitrate nonahydrate, $Al(NO_3)_3$ -9 H_2O and yttrium nitrate hexahydrate, $Y(NO_3)_3$ -6 H_2O . Al_2O_3 preforms with porosity varying from 26 to 50% were obtained after sintering at temperature ranging from 1100 to 1500 °C. Sintering of the infiltrated Al_2O_3 preforms led to formation of YAG particles due to reaction between Y_2O_3 and Al_2O_3 at high temperature. It was found that variation of porosity of alumina preforms and sintering temperature is an effective way to fabricate Al_2O_3 -YAG composite with an unusual combination of properties. Open porosity was in the range 15-35%, specific surface was 0.6-6.1 m^2/g , pore size was 150-900 nm whereas compressive strength was from 50 to 250 MPa. The effect of sintering temperature on YAG formation and phase composition were investigated using X-ray diffractometry whereas microstructure of the composite was analysed by scanning electron microscopy.

Keywords: Al_2O_3 -YAG, Porous ceramics, Composites.

1. Introduction

Good combination of chemical, thermal, mechanical and electrical properties make alumina (Al₂O₃) one of the most frequently used structural materials especially when it comes to industrial application [1]. In general, oxide ceramics have been considered less suitable for high temperature application then non-oxide ceramics due to degradation of mechanical properties at high temperature [2]. On the other side, oxide ceramics, such as alumina, have oxidation and corrosion resistance superior to any other ceramics. Bearing this in mind it is expected that the improvement of high temperature mechanical properties such as strength and fracture toughness will allow oxide ceramics to be effectively used in a wide range of applications at high temperature [2-5]. It is well documented that mechanical properties of alumina can be improved by incorporation of the second phase inclusions into alumina matrix [6, 7]. Yttrium aluminium garnet (YAG) or $3Y_2O_3 \cdot 5Al_2O_3$ is one of the possible reinforcing phases because of its excellent creep resistance, similar coefficient of thermal expansion and no reaction with alumina [8, 9]. There are three crystal phases in the $Al_2O_3 \cdot Y_2O_3$ system, YAG (Y₃Al₅O₁₂), YAP (YAlO₃) and YAM (Y₄Al₂O₉) [10, 11]. Among these phases YAG

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possesses the highest aluminium content and therefore the highest stability in contact with alumina [6-8]. The influence of YAG content ranging from 5 to 30 vol.% on the mechanical properties and microstructure of Al₂O₃-YAG composites was investigated by Lach et al. [6-8]. The highest values of mechanical properties such as fracture toughness, strength and hardness were measured in samples containing 10 vol. % YAG, sintered at 1600 °C. However, there are various applications of oxide ceramics when the components are not exposed to excessive load and therefore superior mechanical properties are not the key demand. It would be important to point out porous ceramics which are of significant interest due to their wide application in high-temperature filtration and absorption, catalysis and catalyst support, thermal insulation, thermal gas separation, lightweight structural as well as thermal structural application [5, 12]. The key properties of porous ceramics used in high temperature environment are high melting point, low thermal conductivity, chemical inertness, open pore structure; especially pore size distribution and decent mechanical properties which allow easy handling and machining without excessive chipping.

There are different routes and different precursors of alumina and Y_2O_3 (yttria) which has been used to fabricate Al_2O_3 -YAG composite. Alumina and yttria powders can be simply mixed by mechanical mixing [13] which can be either dry or wet. This method requires extensive ball milling which usually introduces considerable amount of impurities [9, 13]. Somewhat better homogenization of aluminium and yttrium atoms is usually provided by using water solution of alumina and yttrium precursors. For example, aluminium and yttrium nitrates or chlorides were used in co-precipitation method which is considered as time consuming process [6-9]. Sol-gel technique should also be mentioned as a method of Al_2O_3 -YAG composite preparation. This technique is based on the use of corresponding alcoxides which are usually quite expensive [9]. Palmero et al. prepared Al_2O_3 -5 vol. % YAG composite from commercial α -alumina powders and aqueous solutions of YCl $_3$ ·6H $_2$ O [1, 14]. Different routes of preparing alumina-yttrium system lead to a variety of microstructures with different properties.

In the present work, porous Al₂O₃-YAG composites were fabricated by infiltration of alumina performs with water solution of aluminium nitrate and yttrium nitrate. The technique is based on precipitation of alumina and yttrium precursors within porous alumina compacts. The properties and composition of Al₂O₃-YAG composites were varied by changing the porosity of alumina performs and sintering temperature. According to our knowledge this method has not been investigated by other researchers.

2. Experimental

High purity Al₂O₃ powder (CT3000-SG, Alcoa) was pressed under mechanical pressure of 10 MPa into cylinders with diameter of 16 mm. In order to fabricate alumina performs with different porosity, powder compacts were sintered for 2h at five different temperatures: 1100, 1200, 1300, 1400, and 1500 °C. This heat treatment will be denoted as perform sintering. After the cylinders were vacuumed in vacuum chamber for about 30 minutes they were dripped with water solution of aluminium nitrate nonahydrate, Al(NO₃)₃·9H₂O and yttrium nitrate hexahydrate, Y(NO₃)₃·6H₂O with concentration of 0.38 g/cm³ and 0.23 g/cm³, respectively. The samples were dried at 120 °C for 4 h to remove water and subsequently calcined at 700 °C for 2 h to remove nitrogen. The procedure of vacuuming, dripping, drying and calcination was repeated six times to increase the aluminium and yttrium content in Al₂O₃ performs. Hereafter, this six-step procedure will be called infiltration. The samples were afterwards sintered again for 3 h in order to transform aluminium and yttrium containing compounds into YAG. The temperature of final sintering was equal to the temperature of alumina perform sintering. Powder compacts consisting of commercial Al₂O₃ and Y₂O₃ powders were prepared and sintered for comparative purpose. According to Al₂O₃-

 Y_2O_3 diagram [10, 13], the present fraction of Y_2O_3 in powder compacts is expected to give Al_2O_3 -YAG composite with 15 vol. % YAG. The samples were sintered at 1000 and 1100 °C for 3 h. Density and open porosity of the obtained samples were measured by applying Archimedes' method in xylene and referred to the theoretical density (TD) using 3.97 g/cm³ for alumina [15] and 4.56 g/cm³ for $Y_3Al_5O_{12}$ [16]. The crystalline phases were identified by X-ray diffraction (XRD) analysis using filtered Cu K α radiation (Siemens D5000). Young's modulus was determined by the ultrasonic method (SONIC viewer - MODEL 5210) using following equation [17]:

$$E_{dyn} = V^{2} \cdot \rho \cdot \frac{(1 + \mu_{dyn})(1 - 2 \cdot \mu_{dyn})}{1 - \mu_{dyn}}$$
 (1)

where V is the pulse velocity, ρ is the bulk density (kg/m³), and μ_{dyn} is the dynamic Poisson's ratio. The microstructure analysis was conducted on polished and thermally etched surface by means of scanning electron microscopy (SEM) (JEOL JCM-5800). Prior to the examination the surface was coated with a thin layer of gold. Since the samples were highly porous, the size of individual grains was determined by simple measuring and averaging the grain diameter in vertical and horizontal direction. Open porosity, pore volume, average pore radius as well as specific surface were measured by mercury porosimetry (Porosimeter 2000 Series, Fisions Instruments). Compressive strength measurement was carried out using uniaxial compressive test on Universal testing machine INSTRON 1185.

3. Results and discussion

3.1 Phase identification

Fig. 1 shows the XRD patterns of infiltrated alumina performs sintered at different temperature. The figure indicates that temperature of 1100 °C is sufficient to cause transformation of aluminium and yttrium containing compounds within alumina performs into YAG. As can be seen, the sample sintered at 1100 °C as well as samples sintered at higher temperature consist of Al₂O₃ and YAG. The phase composition of sintered samples is presented in Tab. I. Volume fractions of Al₂O₃ and YAG were calculated in two different ways. The first way is based on the intensity of XRD peaks of sintered samples whereas the second way is based on the increase in mass of alumina performs after infiltration and final sintering. It was assumed that sintering at high temperature completely removes water and nitrogen from infiltrated nitrates while Al, Y and O, present in the residue, finally make YAG.

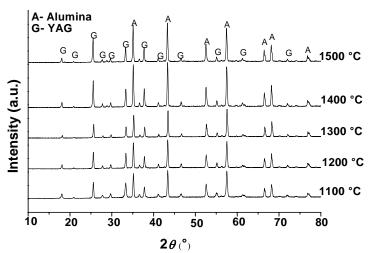


Fig. 1. XRD patterns of samples sintered at temperature ranging from 1100 °C to 1500 °C.

Sintering temperature (°C)	Volume fraction (%)					
	XRD peaks		Mass increase			
	Al ₂ O ₃	YAG	Al ₂ O ₃	YAG		
1100	84.5	15.5	85.4	14.6		
1200	86.3	13.7	86.4	13.6		
1300	90.9	9.1	91.1	8.9		
1400	90.6	9.4	91.6	8.4		
1500	92.1	7.9	95.8	4.2		

Tab. I Volume fraction of Al₂O₃ and YAG in samples calculated from XRD peaks and mass increase.

^{*}sintering temperature of infiltrated alumina preforms is equal to the sintering temperature of alumina preforms

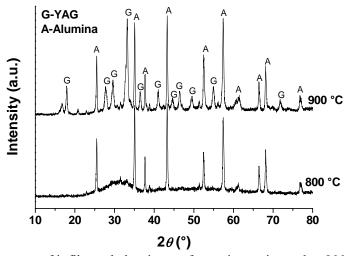


Fig. 2. XRD patterns of infiltrated alumina perform pieces sintered at 800 °C and 900 °C.

Therefore the increase in weight of alumina performs is equivalent to the amount of newly formed YAG. The values presented in Tab. I show a good agreement between the phase compositions determined by two different approaches. It is evident that the fraction of YAG decreases with an increase in sintering temperature. This is the result of decrease in porosity of alumina performs with sintering temperature, which will be discussed latter. As expected, the reduced porosity of alumina compacts obtained at high temperature leads to smaller amount of infiltrated nitrates and therefore smaller fraction of YAG in the sintered composite. As mentioned before, temperature of 1100 °C was sufficient to cause formation of YAG. Now the question arises as what is the lowest temperature at which YAG can be formed. The alumina performs sintered at temperature below 1100 °C were very fragile and they simply fell apart during infiltration. However, the alumina perform pieces were soaked in nitrates solution and thermally treated at temperature below 1100 °C. It was found that YAG crystallizes between 800 and 900 °C (Fig. 2).

Now, one can argue that the same composite material can be obtained by simple sintering of compacts consisting of Al_2O_3 and Y_2O_3 . For this reason compacts of alumina containing amount of Y_2O_3 expected to yield 15 vol. % of YAG were sintered at temperature ranging from 900 to 1100 °C. Volume fraction of YAG of 15% was chosen as that was the largest amount of Y_2O_3 measured in sample obtained by nitrates infiltration. XRD diagram given in Fig. 3 indicates that formation of YAG in samples consisting of commercial alumina and yttrium powders occurs between 1000 and 1100 °C. Even at 1100 °C, there is still small amount of unreacted Y_2O_3 indicating that the infiltration of aluminum and yttrium nitrates in alumina perform can lower the temperature of crystallization of YAG for more than 200 °C.

In addition, small amount of YAM (Y₄Al₂O₉) side product was also detected in samples obtained by sintering of oxides.

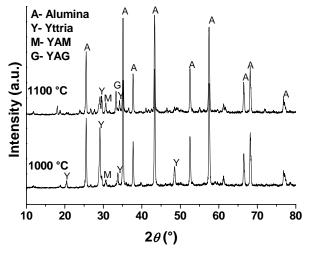


Fig. 3. XRD patterns of alumina/yttrium samples sintered at 1000°C and 1100 °C for 3h.

3.2. Density, porosity and pore characteristics

As mentioned, during the infiltration procedure, the pores of alumina performs were filled with YAG precursor which transformed into YAG during final sintering. From this point of view the composition of YAG precursor is not of great importance and therefore a detail analysis was not conducted. Based on the XRD pattern given in Fig. 2 which shows that YAG precursor is amorphous after heating at 800 °C it is believed that YAG precursor consists of well mixed aluminum, yttrium and oxygen atoms. This disordered structure transforms into crystalline YAG phase during sintering process owing to increased atom mobility and reactivity at high temperature. Fig. 4 describes the effect of sintering temperature on density of alumina performs as well as the effect of infiltration and final sintering on density of the Al₂O₃-YAG composite. As expected, the increase in sintering temperature was an effective way to obtain alumina performs with gradually increasing density, from 1.9 g/cm³ in sample sintered at 1100 °C to 2.93 g/cm³ in sample sintered at 1500 °C. It can also be seen that the six-step infiltration of alumina performs considerably increases the density of samples whereas the effect of final sintering on the increase of density is minimal.

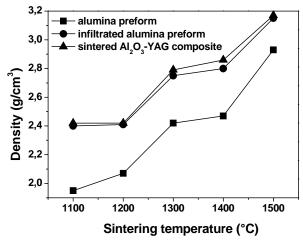


Fig. 4. Effect of temperature on density of alumina performs, infiltrated performs and Al₂O₃-YAG composite.

In order to explain the infiltration process it would be of great importance to analyze the change of open porosity which is presented in Fig. 5. Unlike density, open porosity of alumina performs continuously decreases with an increase in sintering temperature from 50% in perform obtained at 1100 °C to 25% in perform obtained at 1500 °C. As expected, the infiltration reduces porosity since the pores are filled with infiltrated YAG precursor. It is interesting to note that the porosity increases after final sintering despite the fact that the change of density is almost negligible (Fig. 4). This unexpected finding can be explained by the shrinkage of amorphous YAG precursor during sintering. The crystallization of amorphous YAG precursor which fills the pores in alumina perform is accompanied with structure reordering which is followed by shrinkage of YAG precursor and separation of newly crystallized YAG particle from the wall of the pore in alumina perform. Thus, the separation introduces an additional open porosity in Al₂O₃-YAG composite. Finally, the obtained composites have open porosity ranging from 40% in sample sintered at 1100 °C to 20% in sample sintered at 1500 °C. The above mentioned separation of YAG particle from pore wall was also followed by creation of new surface which should have led to an increase in specific surface.

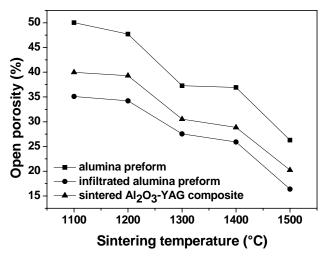


Fig. 5. Effect of temperature on open porosity of alumina performs, infiltrated performs and Al_2O_3 -YAG composite.

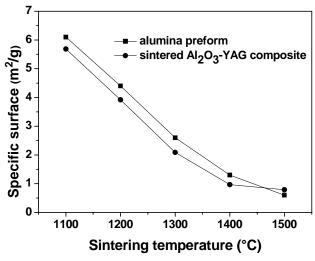


Fig. 6. Effect of temperature on specific surface of alumina performs and Al₂O₃-YAG composite.

Fig. 6 presents the specific surface of alumina performs and Al₂O₃-YAG composite sintered at different temperatures. It is worth noting that at 1500 °C the specific surface of Al₂O₃-YAG composite is larger than that of alumina perform. This means that despite the fact that the performs were infiltrated with YAG and open porosity decreased (Fig. 5) the specific surface of the Al₂O₃-YAG composite increased due to shrinkage of infiltrated material during sintering and consequent separation of YAG particles from alumina wall. According to Fig. 6, it can be concluded that the separation effect is more pronounced at high temperature owing to larger shrinkage of YAG particles. At lower temperature, between 1100 and 1400 °C, the effect of separation is less exaggerated and specific surface of Al₂O₃-YAG composite is just slightly lower than that of alumina perform.

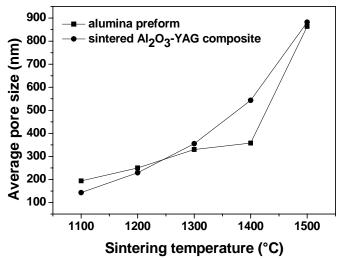


Fig. 7. Effect of temperature on average pore size of alumina performs and Al₂O₃-YAG composite.

It is also important to examine the effect of infiltration and sintering on the average pore size which is presented in Fig. 7. As the figure shows, the average pore size in alumina performs increases with sintering temperature which is expected knowing that high temperature promotes sintering. During the sintering process the number of pores decreases while the average pore diameter increases. In these samples the increase in temperature from 1100 to 1500 °C was followed by increase in the average pore size from ~190 to ~860 nm. The comparison of these values with the values of pore diameter of sintered composite reveals that the average pore diameter slightly changes after infiltration procedure and final sintering. The change is the result of two effects. The first one is the effects of sintering which follows the infiltration process and the second one is the effect of infiltration itself. As mentioned, sintering normally increases pore size whereas infiltration is expected to decrease average pore size as material simply fills the pores of alumina performs. Fig. 7 shows that the effect of infiltration is dominant in samples sintered at lower temperature. The average pore size of alumina performs obtained at 1100 and 1200 °C slightly decreases after infiltration and subsequent sintering. Clearly, temperature as high as 1200 °C was not sufficiently high to allow fast sintering and consequent increase in pore size. Therefore the decrease in pore size is the result of YAG infiltration. On the other side, the pore size in the performs obtained at 1300-1500 °C increases after infiltration and following sintering. In this case sintering process is considerably faster and overall result is the small increase in pore size. This conclusion is also supported by the results presented in Tab. II which shows that shrinkage of sintered composite is considerable higher at temperature above 1200 °C owing to fast sintering process.

Tab. II Shrinkage of alumina performs after infiltration and sintering at different temperature (difference between diameters (r) of alumina performs/cylinders and obtained composites).

Sintering temperature (°C)	1100	1200	1300	1400	1500
Shrinkage (Δr) (%)	0.6	0.9	7.0	7.9	13.3

3.3. Young's modulus and compressive strength

It is well known that porosity strongly affects Young's modulus. The values of Young's modulus, determined by the ultrasonic velocity method, are presented in Fig. 8 which shows that after infiltration procedure Young's modulus of all samples increases due to the decrease in porosity (Fig. 4). Besides Young's modulus, compressive strength is also strongly influenced by porosity.

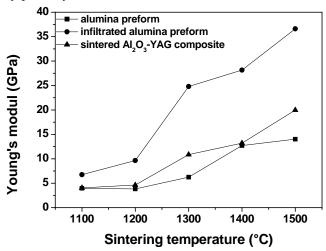


Fig. 8. Effect of temperature on Young's modulus of alumina performs, infiltrated performs and Al₂O₃-YAG composite.

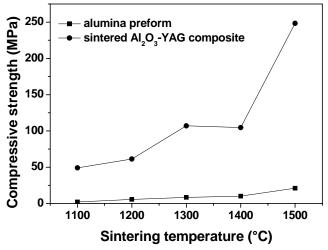


Fig. 9. Effect of temperature on compressive strength of alumina performs and Al₂O₃-YAG composite.

Fig. 9 shows the effect of temperature on compressive strength of alumina performs as well as the strength of composite obtained after infiltration and following sintering. It can be seen that the compressive strength of alumina performs continuously increases with

sintering temperature as the result of the increase in density. Furthermore, the infiltration and subsequent sintering of these performs significantly improve compressive strength. The strength of composite sintered at 1500 °C is about 250 MPa which is considerably higher than that of alumina performs (~ 20 MPa). This is expected knowing that infiltration of YAG decreases porosity, i.e., increases density. Strength (σ_p) and porosity (P) are related by the following empirical relationship [18]:

$$\sigma_n = \sigma_0 e^{-BP} \tag{2}$$

where σ_0 is the strength of the pore free material and B is a constant that depends on the distribution and morphology of the pores. According to the equation the reduction of porosity results in an exponential increase in compressive strength. This kind of relationship was also found in this study. The measured values for compressive strength which are presented in Fig. 9 are fitted to an exponential function (Fig. 10). It was found that the strength of pore free material is ~ 2235 MPa which is within the range of values measured in commercially available alumina ceramics [19].

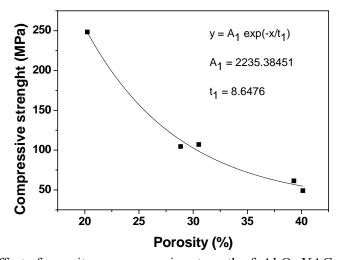


Fig. 10. Effect of porosity on compressive strength of Al₂O₃-YAG composite.

3.4. Microstructure

Fig. 11 shows the scanning electron microscopy (SEM) images of Al_2O_3 -YAG composite obtained at different temperatures. As can be seen, the average grain size continuously increases with sintering temperature from bellow 1 μ m in sample sintered at 1100 °C to ~ 3 μ m in sample sintered at 1500 °C. Again, this is quite expected bearing in mind that high temperature promotes sintering and therefore grain growth. In samples sintered at 1200, 1300 and 1400 °C the size of spherical grains is in the range of 0.2-2.0, 0.3-2.5 and 0.4-4.5 μ m, respectively. Although it is evident that sintering takes place in these samples, the grains are still weakly bonded. At higher temperature such as 1500 °C the grains are much larger (0.7-7 μ m) and bonded more rigidly which resulted in considerable increase in compressive strength (Fig. 9).

Although the above SEM images were very useful in determining the size and morphology of grains it was not possible to distinguishing alumina grains from YAG grains. Therefore the composites sintered at 1200 and 1500 °C were analysed by SEM using backscatter mode (BSE) and electron dispersive spectroscopy (EDS). Fig. 12a shows the SEM-BSE image of composite sintered at 1200 °C. Again, it was quite difficult to distinguish alumina from YAG as the YAG grains were very small. Since the average pore diameter of alumina performs obtained at 1200 °C was ~ 250 nm, it reasonably to assume that YAG grains are smaller than that due to the shrinkage of infiltrated material during subsequent sintering. The relatively large, white grain located in the center of the image was analysed by

EDS. The EDS spectrum, given in Fig. 12b, reveals that this grain is YAG as it contains Y, Al and O. The presence of gold (Au) in EDS spectrum comes from thin layer of gold coating. This finding suggests that YAG phase in Al₂O₃-YAG composite should be white or at least brighter than alumina. SEM micrograph and yttrium mapping micrograph of sample sintered at 1500 °C are given in Fig. 13. Careful comparison of SEM image (Fig. 13a) and image of yttrium distribution (Fig. 13b) allows identification of YAG phase. It appears that submicron YAG grains are located among large alumina grains (arrows in Fig. 13a). This is expected knowing that pores in alumina performs obtained at 1500 °C were ~850 nm in diameter. It is evident that the size of YAG grains is close to the size of pores in alumina performs simply due to limited space available for infiltration of YAG precursors.

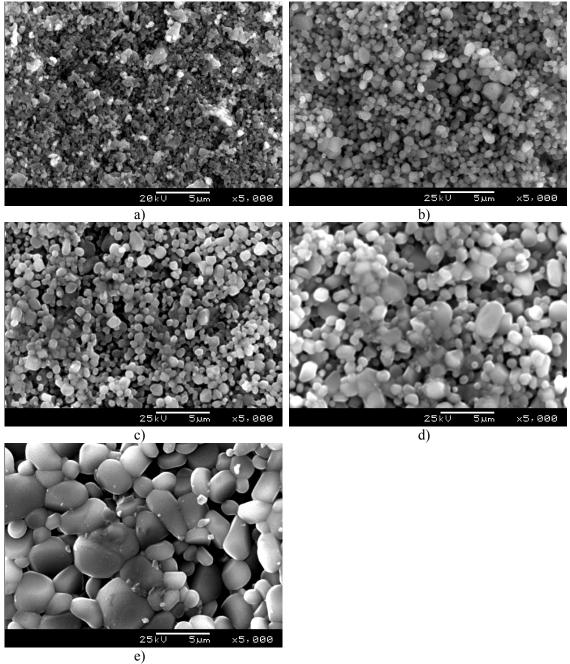


Fig. 11. SEM images of Al₂O₃-YAG composites obtained at a) 1100 °C; b) 1200 °C; c) 1300 °C; d) 1400 °C and e) 1500 °C.

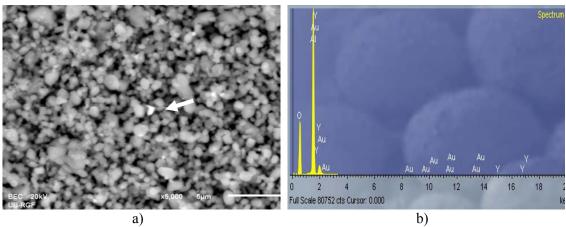


Fig. 12. a) SEM-BSE image and b) EDS spectrum of composite sintered at 1200 °C.

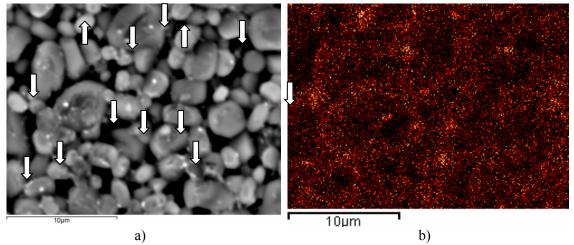


Fig. 13. a) SEM-BSE image and b) YK α (yttrium) mapping images by SEM-EDS method of sample sintered at 1500 °C.

4. Conclusions

Porous Al_2O_3 -YAG composites were prepared by infiltration and precipitation method. The volume fraction of YAG continuously increased from 4 vol.% in sample sintered at 1500 °C to 15 vol.% in sample sintered at 1100 °C. Complete YAG crystallization was achieved at all given sintering temperatures. Crystallization was followed by shrinkage of YAG precursor and separation of newly crystallized YAG particles from the wall of the pores in alumina performs which led to fabrication of material with interesting combination of properties. The porosity decreased from 40 vol.% in sample sintered at 1100 °C to 20 vol.% in samples sintered at 1500 °C. At the same time, specific surface decreased from 6 m²/g to below 1 m²/g. On the other side, average grain size continuously increased from below 1 µm in sample sintered at 1100 °C to 7 µm in sample sintered at 1500 °C. The highest Young's modulus of 20 GPa and the highest compressive strength of 250 MPa were measured in composite sintered at 1500 °C.

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Садржај: Al_2O_3 -YAG композит добијен је синтеровањем порозних Al_2O_3 узорака (предформи) претходно инфилтрираних воденим раствором алуминијум нитрата нонахидрата $Al(NO_3)_3$ · $9H_2O$ и итријум нитрата хексахидрата $Y(NO_3)_3$ · $6H_2O$. Порозност Al_2O_3 предформи добијених синтеровањем у температурном опсегу од 1100 до 1500 °C је варирала од 26 до 50%. Синтеровање инфилтрираних Al_2O_3 предформи је довело до формирања YAG честица услед реакције између Y_2O_3 и Al_2O_3 на високим температурама. Утврђено је да се варијацијом порозности Al_2O_3 предформи

и температуре синтеровања може добити Al_2O_3 -YAG композит са необичном комбинацијом особина. Отворена порозност је била у опсегу 15-35%, специфична површина 0.6- $6.1 \text{ m}^2/\text{g}$, величина пора у опсегу 150-900 пт, док је компресиона чврстоћа била у распону од 50 до 250 MPa. Утицај температуре синтеровања на формирање YAG и фазни састав испитани су рендгено-дифракционом анализом, док је микроструктура композита анализирана скенирајућом електронским микроскопијом. **Кључне речи:** Al_2O_3 -YAG, порозна керамика, композити.

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